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RESEARCH MEMORANDUM

INFILTRATION OF TITANIUM CARBIDE WITH SEVERAL METALS

By Raymond S. Gurnick and Anthony L. Cooper

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Cleveland, Ohio

NATIONAL ADVISORY COMMITTEE
FOR AERONAUTICS
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SUMMARY

An investigation was made to determine the applicability of infil-
tration techniques for producing ceramals of titanium carbide with various
metals or alloys. Nickel, S-816, Stellite 21 (AMS-5385), Hastelloy B,
and Stellite 93 were used as infiltrant materials.

Specimens for elevated-temperature modulus-of-rupture and stress-
rupture testing, and turbosupercharger blades, were fabricated by infil-
trating titanium carbide with S-816 alloy. Turbosupercharger blades were
also fabricated by arc-welding or precision-casting of alloy roots to
infiltrated ceramal airfoils by means of titanium carbide and S-816 alloy.

The ceramal composed of titanium carbide and S-816 had a modulus-
of-rupture strength of more than 100,000 pounds per square inch at 1600° F
and 54,000 pounds per square inch at 1800° F. The stress-rupture life was
1082 hours at 1500° F and 112 hours at 1600° F at a stress of 15,000 pounds
per square inch.

INTRODUCTION

The desire to decrease the amount of strategically critical metals
used in the gas-turbine engine and to increase the operating temperature
has led to the investigation of ceramic and ceramal materials for possible
turbine-blade application. These materials are believed to be suitable
for such use because of high strength-to-weight ratios and good elevated-
temperature properties (references 1 and 2).

One of the purposes of this study was to devise techniques for
infiltrating a refractory ceramic skeleton with a heat-resisting alloy
to produce a ceramal. If this procedure proved feasible, the end product
would be a material in which the defects associated with the large
shrinkages and with the residual pores encountered in the more conventional
methods of powder fabrication are eliminated.

This method has been used to produce gas-turbine compressor stator
blades (reference 3) by infiltrating an iron skeleton with a liquid copper
alloy. Two applications of the infiltration technique have been considered
for producing heat-resisting materials. In one application, skeletons
of the refractory metals tungsten and molybdenum were infiltrated with

nickel or cobalt base alloys (reference 4). In the other, refractory
metal skeletons were infiltrated with low-melting ceramics (reference 5).

The ceramic chosen for this investigation was titanium carbide
because titanium carbide base ceramals have good high-temperature proper-
ties (references 2 and 6). A preliminary study (reference 4) has shown
that titanium carbide is promising as a skeleton material for infiltration
with various heat-resisting alloys as infiltrant materials.

Preliminary high-temperature strength properties were obtained to
permit a comparison to be made with titanium carbide base ceramals
fabricated by conventional hot-pressing and cold-pressing
techniques.

The lack of ductility and resulting notch sensitivity of ceramal
turbine blades present the problem of retaining the blades in a turbine
wheel during operation. It was felt that a composite blade having a
ceramal airfoil and a ductile alloy root would eliminate much of this
difficulty. Therefore, an auxiliary purpose of this program was to
attempt to produce such a body; the infiltration technique was utilized
to make the ceramal airfoil and standard casting and welding processes
were used to affix an alloy root to it.

MATERIALS

The titanium carbide powder used in the preliminary investigation
had an average particle size of 1 to 2 microns. The chemical analysis
was as follows:

Element	Percent by weight	
	Analysis	Stoichiometric
Titanium	79.65	79.95
Carbon (combined)	18.82	20.05
Carbon (free)	.38	-----

The nickel metal used was reported by the supplier to contain
99.9 percent nickel and had a powder particle size of -300 mesh.

The nominal analyses of alloys used as infiltrant materials are
shown in the following table:

Alloy	Nominal analysis (percent by weight)							
	Co	Ni	Fe	Cr	Mo	W	Cb	Form
S-816	bal	20.0	3.0	20.0	4.0	4.0	4.0	wrought
Stellite 21	bal	2.5	2.0	30.0	5.5	---	---	cast
Hastelloy B	---	bal	5.5	----	30.0	---	---	cast
Hastelloy C	---	bal	5.5	16.5	17.0	4.0	---	cast
Stellite 93	6	---	bal	17.0	16.0	---	---	cast

EVALUATION PROCEDURE

Density. - Densities of the porous titanium carbide skeletons were calculated from the measured weights and volumes of machined blocks. For the infiltrated bodies, the method of differential weighing in air and water was used.

Modulus of rupture. - Rectangular modulus-of-rupture specimens approximately $\frac{1}{2} \times \frac{5}{16} \times 3\frac{1}{4}$ inches were prepared by finish-grinding the infiltrated bars. Tests were conducted at 1600° and 1800° ±10° F by means of the general apparatus and procedure of reference 2. The specimen was placed in a furnace operating at the test temperature and was supported on two silicon carbide knife edges spaced $2\frac{1}{2}$ inches apart. Loading was accomplished at the center through a third opposing knife edge. Ten minutes were allowed for the specimen to reach the test temperature. The specimen temperature was measured at the midpoint of the specimen with a chromel-alumel thermocouple. A nominal loading rate of 2000 pounds per square inch per minute on the extreme outer fiber at the specimen midpoint was used.

Stress rupture. - Stress-rupture specimens as shown in figure 1 were evaluated at 1500° and 1600° ±10° F at a stress of 15,000 pounds per square inch by means of the general apparatus and procedure of reference 7. The specimen was held in grips of Inconel X having sufficient plasticity at or near the test temperature to assume axial alinement. A motor-driven gear train actuated by limit switches provided an automatic take-up of specimen and pull-rod elongation to maintain a level loading beam. A preload of 2000 pounds per square inch was then applied. The furnace was placed in position and the temperature raised to the test temperature. The specimen was then "soaked" for one hour at the preload stress. The total load was finally applied in increments small enough to permit the take-up mechanism to function efficiently. Temperature was measured by three thermocouples located at the top, bottom, and center of the specimen gauge length.

Turbine tests. - Turbosupercharger blades were evaluated in gas-turbine wheels of approximately $12\frac{1}{2}$-inch total diameter by means of the equipment and general procedure of reference 6. (The turbosupercharger was used as a small-scale turbine test rig.) A small gas turbine supplied with hot gases from a turbojet combustion chamber was used to evaluate the performance of the turbine blades. The turbine operating temperatures were indicated by thermocouples that measured gas temperature in the inlet duct 12 inches upstream of the turbine inlet. After combustion was initiated, operating conditions were achieved in approximately 3 to 5 minutes. The wheel was then operated with no load applied at the shaft at 26,000 rpm and an inlet gas temperature of $1650^{\circ} \pm 15^{\circ}$ F until blade failure occurred.

RESULTS

Since a major portion of the investigation involved devising new fabrication techniques, these techniques constitute one very important result of the study and will be presented in some detail.

Discussion of Fabrication Procedures

Titanium carbide skeletons. - Titanium carbide skeletons were hot-pressed for 1 hour at a load of 2000 pounds per square inch in 2-inch graphite dies heated by induction. The temperature was varied from 2400° to 3100° F in 100° increments. The technique used was that described in reference 8. These runs yielded skeleton blocks having from 24 to 40 percent porosity. Tests were then conducted to determine the optimum degree of porosity for infiltration. For these determinations, nickel metal was used as the infiltrant. Infiltration was carried out in a tungsten resistance furnace in a vacuum of 3 to 5 microns. Microscopic examination showed that skeletons having porosities ranging from 32 to 38 percent were uniformly filled and pore-free after infiltration. These bodies were hot-pressed at 2500° F. Those bodies which had porosities less than 32 percent were incompletely filled. Those which had porosities greater than 38 percent were unsatisfactory because the liquid infiltrant was not uniformly retained; distortion and sweating resulted.

A double-acting graphite die was used to hot-press titanium carbide skeletons $3\frac{1}{4} \times 1\frac{3}{4} \times 3/4$ inches in size. In order to reduce the weight of the longer die required to make skeletons of this size, the die wall thickness was decreased; this decrease limited the maximum pressure to 1600 pounds per square inch. Despite the change in pressure from 2000 to 1600 pounds per square inch, the porosity of the titanium carbide skeletons produced at 2500° F was within 2 percent of the porosity obtained in the tests with the 2-inch graphite dies at the same temperature. These skeletons were found to be satisfactory for infiltration and were readily machinable.

Production of shapes. - Modulus-of-rupture and flat, tapered stress-rupture bars were cut from the skeleton blocks on a band saw. The specimens were then smoothed on a belt sander to the required dimensions which were approximately 2 percent oversize to allow for shrinkage in the infiltration operation. Unshrouded turbosupercharger blades and airfoil sections were hand-worked to the desired shape with files and alundum abrasives. Figure 2 shows the final shape of a blade blank prior to infiltration. Figure 3 shows the three basic shapes after infiltration; the test bars have been ground to test dimensions.

Infiltration. - Nickel, S-816, Stellite 21, Hastelloy B, and Stellite 93 were evaluated as infiltrant materials. Infiltration tests were carried out at temperatures $75^\circ \pm 25^\circ$ F above the melting points of the alloys in a vacuum of 3 to 5 microns by induction heating with a tungsten susceptor. Figure 4 shows the structures obtained. In order to limit the scope of the investigation, it was decided to select a single alloy for further study. Since all microstructures appeared sound, S-816 alloy was selected as the infiltrant material on the basis of elevated-temperature strength characteristics.

It also was decided to study the effect of various furnace atmospheres on the infiltration process with titanium carbide and S-816 alloy. The atmospheres used were those that resulted from induction-heating graphite and tungsten susceptors in vacuum, in helium with 8 percent hydrogen, and in argon. Whenever graphite was used as a susceptor, the atmosphere was contaminated with carbon monoxide formed by out-gassing from the graphite. The only atmosphere that consistently yielded dense, completely filled bodies after infiltration was that which resulted when a tungsten susceptor was used and the furnace chamber was evacuated to 3 to 5 microns. This confirmed the work of Meerson (reference 9) who found that a hydrogen atmosphere was detrimental to the infiltration of titanium carbide with cobalt metal and his data also suggest that the presence of carbon monoxide would result in incomplete filling of the titanium carbide skeleton.

The final procedure that was selected for use in producing shapes for physical evaluation consisted in suspending titanium carbide skeletons in a vacuum induction furnace with a tungsten susceptor as shown in figure 5. A quantity of infiltrant calculated to fill the skeleton pore volume was placed on top of the shape. The induction furnace chamber was evacuated to 3 to 5 microns of mercury pressure read on an ionization gage. The temperature was then raised to $75^\circ \pm 25^\circ$ F above the melting point of the infiltrant. As infiltration progressed, the gradual absorption of the liquid infiltrant by the porous skeleton and a progression of color change vertically down the length of the specimen were observed. (This in all probability represented the course of the infiltrant through the skeleton.) When both of these processes were completed, the specimen was furnace-cooled to room temperature under the same vacuum. The time necessary to accomplish filling varied from 15 to 45 minutes depending

on specimen size. When excess infiltrant was used, the liquid sweated
out at the bottom of the body, and when the titanium carbide skeleton
was not properly alined within the furnace, warpage occurred as shown in
figure 6.

Composite blades. - Two methods of forming composite blades were
investigated: (1) simultaneous casting of the root and infiltration of
the airfoil skeleton, and (2) welding of an individually fabricated
metal root to an infiltrated ceramal airfoil.

(1) Casting. Precision investment molds were prepared by the lost
wax process with various commercial mold materials. Since it was neces-
sary to cool the investment molds to room temperature after curing in
order to insert the titanium carbide skeleton into the airfoil cavity,
materials were selected which would not crack or break up when cooled
from the curing temperature. The investment materials used were Ransom
and Randolph No. 711G with a Coatwell coat liquid and a precoat of
aluminum oxide and silicon oxide. Four basic mold designs were used
for trials to produce a composite blade (fig. 7). After the molds were
prepared, the wax was burned out and curing took place by heating to
1500° F at a rate of 100° F per hour and cooling back to room temperature
at the same rate. Titanium carbide skeleton shapes were then inserted
in the mold airfoil cavities as shown in figure 7. S-816 alloy was used
as the infiltrant.

When mold 7(a) was used, a weighed charge of infiltrant, sufficient
to fill the pores of the skeleton and also the root cavity, was placed
at the top of the mold as shown. The mold was then placed in a vacuum
induction furnace and the heating schedule previously described for the
production of continuous infiltrated shapes was used. This method proved
unsatisfactory. There was little or no filling of the root cavity.
Since the volume of infiltrant necessary to fill the root cavity had
to pass through the skeleton, the filling of the interconnecting pores
effectively blocked the excess infiltrant. The airfoil was completely
infiltrated, however.

With mold 7(b) an excess metal charge was placed in the sprue-feed
cavity. The same heating schedule was again employed. It was found that
the thin walls in the underfeed section of the mold were eroded by the
liquid infiltrant, contaminating and causing incomplete filling of the
titanium carbide airfoil.

When mold 7(c) was used, it was found that the liquid infiltrant
flowed preferentially into the airfoil cavity between the airfoil and
cavity wall because of the head of metal above the skeleton. This eroded
the investment material thus destroying the airfoil surface finish.

A single run was made with Stellite 21 as the infiltrant in a fer-
rolite mold of design 7(c). It was found that a metal button could be
affixed to the infiltrated titanium carbide airfoil during infiltration
(fig. 8(a)). Radiographic examination showed no defects. Figure 8(b)
shows a typical area of the ceramal-alloy interface. It can be seen
that a film has remained between the Stellite 21 and the infiltrated
ceramal after infiltration.

With mold 7(d) a previously cast S-816 alloy root and riser were
invested prior to infiltration to form part of the mold. Complete filling
of the titanium carbide airfoil skeleton and root cavity took place.
Figure 9(a) shows an S-816 root insert with blind riser and the composite
turbosupercharger blade after infiltration. It can be seen that the
volume of liquid infiltrant necessary to fill the pores of the airfoil
skeleton has been supplied by the blind riser. Figure 9(b) shows a typi-
cal area of the ceramal-alloy interface for the composite blade. A zone
of porous defective material can be observed. As the titanium carbide
was in contact with the molten infiltrant at the interface for the
longest period of time, this defective zone is probably due to excessive
solubility and erosion at the ceramal-alloy interface.

Although all mold runs were not completely satisfactory, it is
felt that, at least in the light of results from molds 7(c) and 7(d),
further study of the variables involved should result in the production
of sound composite bodies.

(2) Welding. Arc-welding of the titanium carbide - S-816 ceramal
to S-816 alloy was investigated in argon, helium, and argon-hydrogen
atmospheres. The infiltrated material exhibited good weldability under
all three conditions. The composite blade shown in figure 10(a) was
welded in argon-hydrogen with S-816 filler rod. A post-heat cycle was
used to suppress thin-section cracking. Radiographic examination showed
no indication of cracks. Figure 10(b) shows the weld interface structure
and it can be seen that the weld is sound and uniform. Comparison of
the alloy-ceramal interfaces formed by arc-welding and investment casting
(figs. 9(b) and 10(b)) show that the arc-welding method of making a
composite shape gave the more uniform, defect-free bond between the
alloy and the ceramal where S-816 alloy was used.

Preliminary Strength Data

Modulus-of-rupture and stress-rupture bars and turbosupercharger
blades of the type shown in figure 3 were made by infiltrating titanium
carbide with S-816 alloy. The bodies were 50 percent by weight alloy
and had a density of 6.0 grams per milliliter.

Modulus of rupture. - Evaluations were conducted at 1600° and 1800° F with a nominal loading rate of 2000 pounds per square inch per minute on a $2\frac{1}{2}$-inch span. Results were as follows:

Specimen number	Evaluation temperature (°F)	Modulus-of-rupture strength (psi)	Remarks
M-1	1600	> 100,000	reached capacity of apparatus without breaking
M-2	1600	> 100,000	reached capacity of apparatus without breaking
M-3	1800	53,000	
M-4	1800	52,000	
M-5	1800	57,000	

These values are consistently higher than values reported on several titanium carbide base ceramals of cobalt, tungsten, and molybdenum (reference 2).

Stress rupture. - Stress-rupture specimens were tested at 1500° and 1600° F at a stress of 15,000 pounds per square inch. The ceramal had a life of 1082 hours at 1500° F and a life of 112 hours at 1600° F before failure occurred. These values compare favorably with data for ceramal compositions of 62 percent TiC, 30 percent Ni, and 8 percent CbC·TaC·TiC solid solution; this type is currently used for turbine-blade evaluation and has a 100-hour life at 1600° F at a stress of 15,000 pounds per square inch and a 1000-hour life at 1500° F at a stress of 16,000 pounds per square inch.

Turbine tests. - The test conditions in the gas-turbine rig gave an estimated blade temperature of 1600° F. A blade of continuous ceramal composition (fig. 3) failed after 2.5 hours at 26,000 rpm. The stresses in the center of the airfoil of the blade at this speed correspond to those at the center of the airfoil of a turbine blade in the J-33 engine operated at rated speed. Failure occurred in the airfoil section approximately 1/2 inch above the root. Visual examination of the fractured surface indicated a break of the type observed in tensile failures of brittle materials.

An arc-welded composite blade of the type shown in figure 10(a) failed after 1.4 hours at 26,000 rpm and 1600° F estimated blade temperature. Failure occurred in the ceramal material approximately 1/16 inch above the ceramal alloy interface. No evidence of porosity was observed on the fractured surface.

An investment-cast composite blade of the type shown in figure 9(a) failed during acceleration to test conditions. Failure occurred in the defective zone adjacent to the ceramal-alloy interface as shown in figure 9(b).

The performance of the turbine blades in the turbine test fell short of predicted life based on the stress-rupture properties of this material. Further studies are needed to produce turbine blades having the same uniform physical properties as the test bars which were evaluated in the preliminary investigation.

SUMMARY OF RESULTS

1. Hot-pressed titanium carbide skeletons of 32 to 38 percent porosity were infiltrated with nickel, S-816, Stellite 21, Hastelloy B, and Stellite 93 in a vacuum of 3 to 5 microns. The structures obtained were uniform and free of defects.

2. Modulus-of-rupture and stress-rupture test bars and turbosupercharger blades were fabricated by infiltrating shaped titanium carbide skeletons with S-816 alloy. The infiltrated bodies had a density of 6.0 grams per milliliter and a composition of 50 percent by weight alloy.

3. The ceramal composed of titanium carbide and S-816 had a modulus-of-rupture strength of more than 100,000 pounds per square inch at 1600° F and 54,000 pounds per square inch at 1800° F.

4. The ceramal composed of titanium carbide and S-816 had a stress-rupture life of 1082 hours at 1500° F and 112 hours at 1600° F at a stress of 15,000 pounds per square inch.

5. Composite turbosupercharger blades were fabricated from titanium carbide and S-816 alloy by (a) simultaneous casting of the metal root and infiltration of the titanium carbide airfoil skeleton and (b) welding of an individually fabricated metal root to an infiltrated ceramal airfoil. These blades were evaluated in a turbine at a temperature of 1600° F and at a speed of 26,000 rpm. The stresses in the center of the airfoil of the blade at this speed correspond to those at the center of the airfoil of a turbine blade in the J-33 engine operated at rated speed. The maximum life obtained was with the continuous ceramal composition and was 2.5 hours.

CONCLUDING REMARKS

The infiltration technique as a method of fabricating sound bodies from refractory materials has been demonstrated in the laboratory. It has been shown that ceramic bodies can be made which can be readily

shaped prior to infiltration with a molten metal phase. Because of low
shrinkage, these bodies have little tendency to crack during
sintering.

The physical properties obtained in the temperature range investi-
gated can be favorably compared with those of almost all titanium carbide
base ceramals made by conventional powder-metallurgy techniques.

This investigation was preliminary in scope and further studies
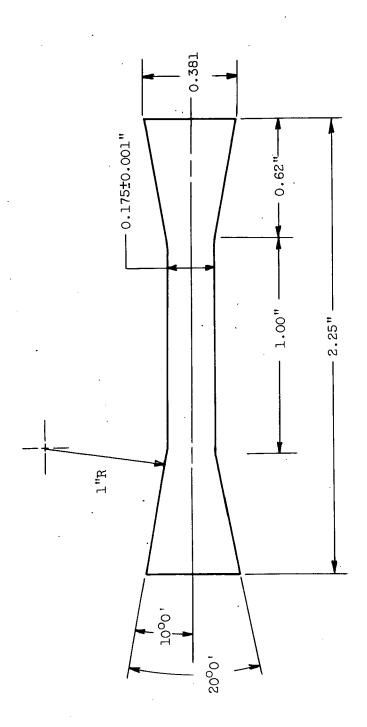are considered necessary to evaluate materials made by this process for
turbine-blade applications.

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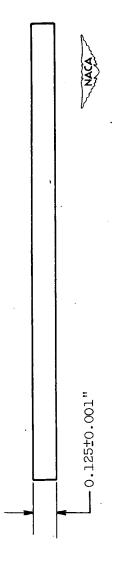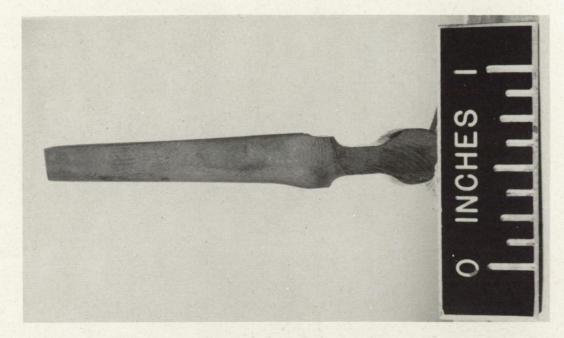   (Trans. from Zhurnal Prikladnoi Khimii, vol. 13, no. 1, 1940,
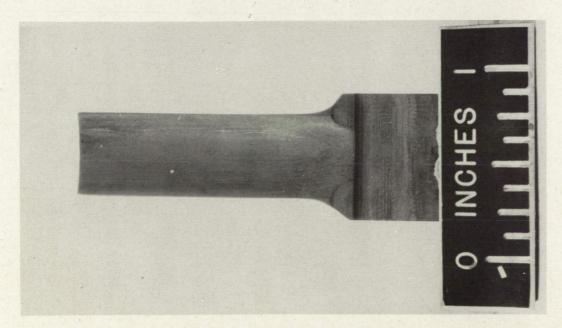   pp. 66-75.)

Figure 1. - Stress-rupture specimen.
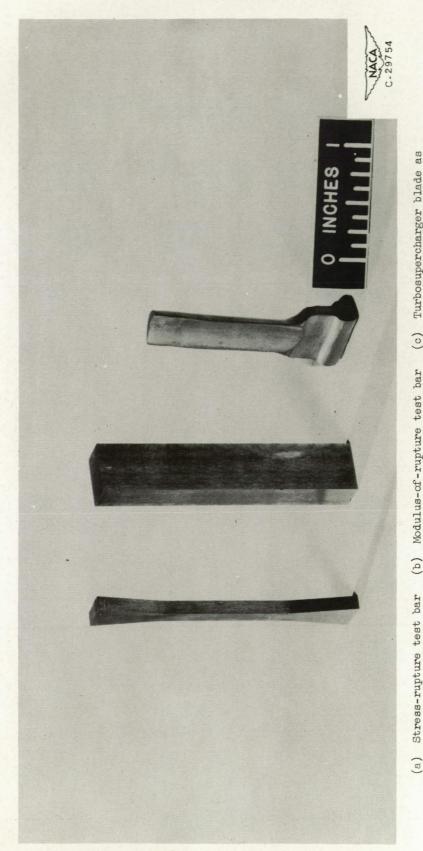
Figure 2. - Blade blank machined from partly sintered titanium carbide.

(a) Stress-rupture test bar   (b) Modulus-of-rupture test bar   (c) Turbosupercharger blade as as ground.   as ground.   infiltrated.

Figure 3. - Specimen and blade shapes after infiltration of titanium carbide with S-816 alloy.

(a) Nickel.

(b) S-816 alloy.

(c) Stellite 21.

(d) Hastelloy B alloy.

(e) Stellite 93 alloy.
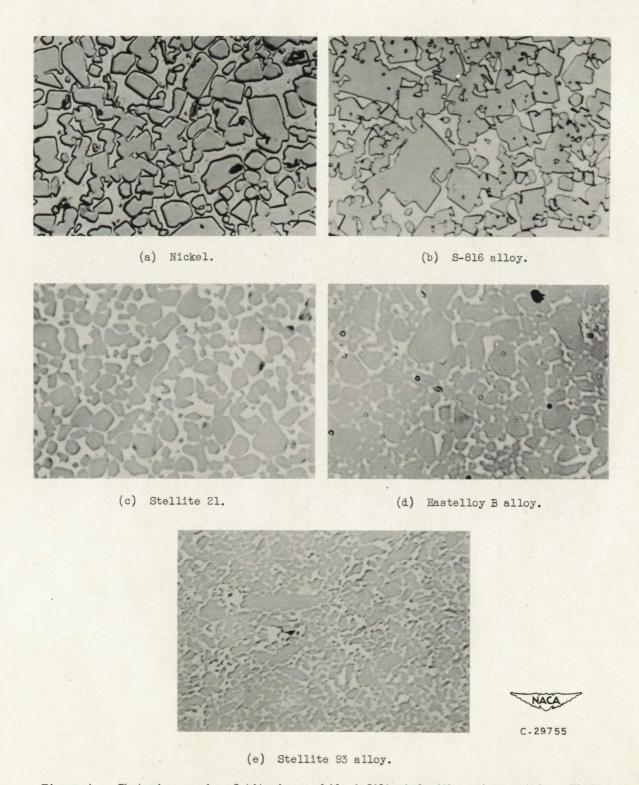
Figure 4. - Photomicrographs of titanium carbide infiltrated with various metals. X1000.

Tungsten
wire
hanger

Infiltrant
metal

4 Alundum spacer rods

0.050" Tungsten
susceptor coil

Alundum crucible
8"×2"×1/8"

Milled zircon

Partly sintered
titanium
carbide blade

Zircon liner
Silica binder

Tungsten
block

1/4" Copper tubing
for induction
heating

Zircon brick
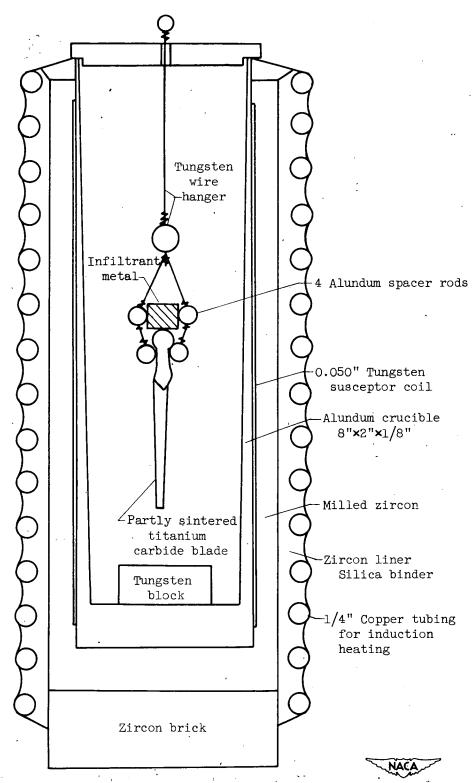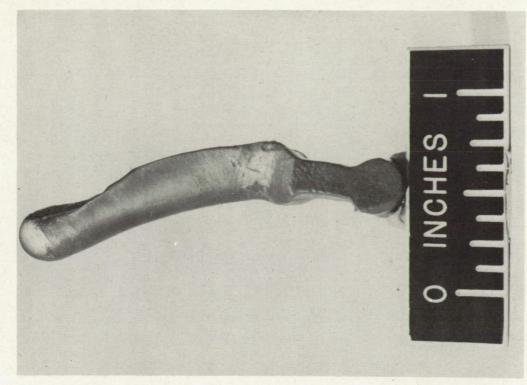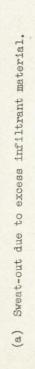
Figure 5. - Induction furnace, crucible, and holder for
infiltration.

(a) Sweat-out due to excess infiltrant material.   (b) Warpage due to excess infiltrant material and nonaxial alinement.
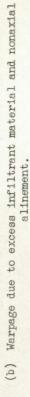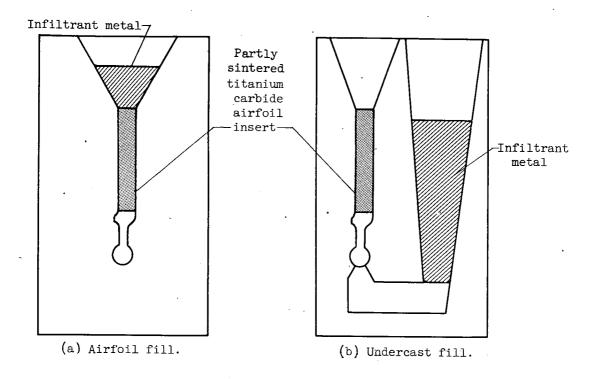
Figure 6. - Effect of excess infiltrant material after infiltration of titanium carbide with S-816 alloy.

Infiltrant metal

Partly
sintered
titanium
carbide
airfoil
insert

Infiltrant
metal

(a) Airfoil fill.

(b) Undercast fill.

Infiltrant metal

Partly sintered
titanium
carbide
airfoil
insert

Precast
metal root
and riser

(c) Root fill.

(d) Modified undercast
fill from blind riser.
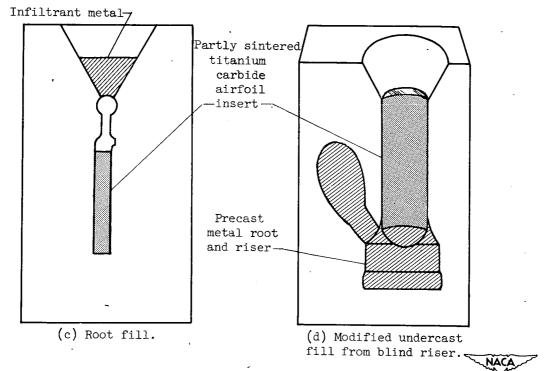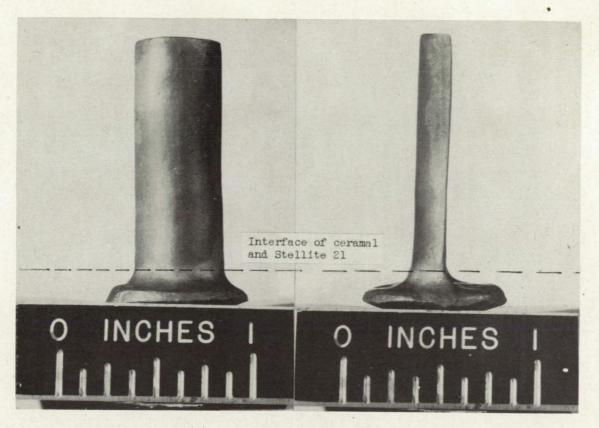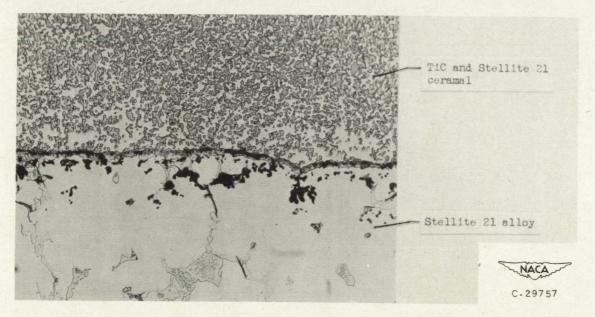
Figure 7. - Mold designs for infiltration casting of composite turbosuper-
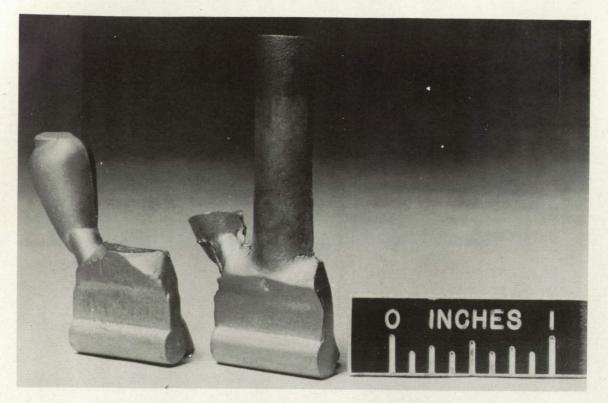charger blades having ceramal airfoils and alloy roots.
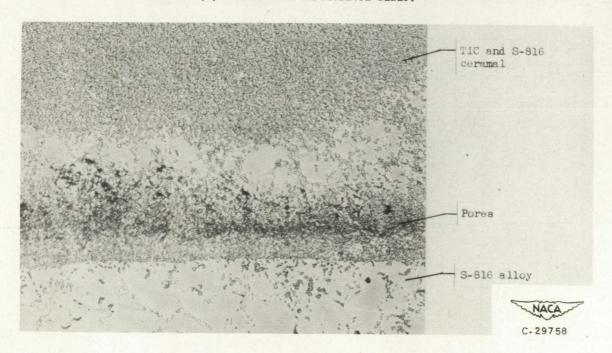
(a) Infiltrated airfoil as cast.

(b) Photomicrograph of interface. Etchant, 5-percent aqua regia; X250.

Figure 8. - Investment-cast composite turbosupercharger blades after infiltration of titanium carbide with Stellite 21 alloy.
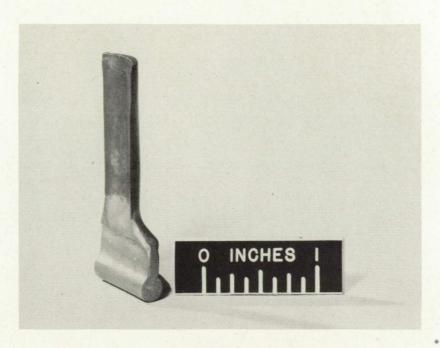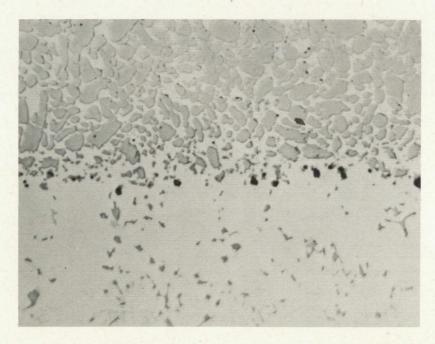
(a) Insert and infiltrated blade.

(b) Photomicrograph of interface. Unetched; X100.

Figure 9. - Investment-cast composite turbosupercharger blade after infiltration of
titanium carbide with S-816 alloy.

(a) Full view.

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(b) Photomicrograph of weld interface. Unetched; X1000.

Figure 10. - Welded composite turbosupercharger blade having infiltrated ceramal
airfoil and S-816 root.